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For: HEAT SHRINKABLE POLYESTER FILM

**Mail Stop AF**

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CERTIFICATE OF TRANSLATION

Dear Sir:

The undersigned, Takeshi Komatani, Ph.D., certifies the following.

1. I am knowledgeable of both Japanese and English.
2. I translated Japanese Patent Application No. 50-122601 into English.
3. The attached document is a true and accurate translation of Japanese Patent Application No. 50-122601.

Respectfully submitted,

Date: 11<sup>th</sup> August, 2008

Takeshi Komatani

(Translator's Name)



(Translation)

Patent Application

October 11, 1975

To: Hideo SAITO, the Commissioner of the Patent Office

1. Title of the Invention:

POLYESTER FILM HAVING EXCELLENT PROCESSABILITY

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| (1) Specification          | 1 |
| (2) Subcopy of the request | 1 |
| (3) Drawing                | 1 |

## SPECIFICATION

### 1. Title of the Invention:

POLYESTER FILM HAVING EXCELLENT PROCESSABILITY

### 2. Claim:

A polyester film having excellent processability obtained by melt-extruding: a polyester consisting of a residue of dibasic acids wherein at least 80% by mol of the dibasic acids is terephthalic acid and a residue of glycols; a block copolyester consisting of a polyester segment having a high melting point and a soft polymer segment having a low melting point, said soft polymer segment having a low melting point being contained in an amount of 0.5 to 10% by weight on the basis of the whole weight of the polymer mixture; and a sulfonic acid metal salt derivative having a heat resistance such that the initial temperature showing a substantial decrease of the weight is 200°C or higher when it is heated in air, said sulfonic acid metal salt derivative being contained in an amount of 0.05 to 5% by weight with respect to the whole weight of the polymer mixture, and subsequently drawing at least uniaxially and then heat-treating the resultant at a temperature of 170°C or higher and lower than the melting point of the polyester.

### 3. Detailed Description of the Invention:

The present invention relates to a polyester film having excellent flexing resistance, pinhole resistance, printability and antistatic properties with low dependence on humidity.

As well known, there has been increasing new desire in the use of polyester films for packaging of foods and industrial parts, protecting films, or the like, since they have a high

crystallizability and excellent transparency, mechanical properties, chemical resistance and heat resistance. However, when the films are used for these packagings, severe burden is given to the films in many cases, and in such a case, the existing polyester films have various defects. For instance, in vacuum packaging, stress tends to concentrate on sharp portions of the corners of a packaging bag, and as a result, pinholes are formed therein and microorganisms get in through the pinholes. Moreover, in a retort packaging, the film is shrunk or deteriorated during the sterilization treatment at high temperature, and thereby the film becomes brittle. In the case where the film does not have water resistance, the film is more likely to break. In frozen food packaging, the package has many sharp corners since the contents thereof are frozen and the contents are hardly deformed and therefore the package is significantly injured. In printing process, many printing companies have desired to print with ink for cellophane from economical and qualitative viewpoints, but normal polyester films have a defect that ink for cellophane is difficult to adhere thereto. Furthermore, since polyester resins have high electrical insulation properties, the films produced therefrom are very likely to be electrically charged, which causes various troubles due to the occurrence and the accumulation of static electricity. Particularly, under a low humidity environment, the occurrence of the static electricity becomes larger, which is a major cause of the lowering of the workability and the value of the products.

It is very difficult to improve even one of these defects, that is, inferior pinhole resistance, flexing resistance, printability and antistatic properties, and it is more difficult to give these properties to a polyester film at the same time. For example, in order to give toughness such as flexing resistance and pinhole resistance to a macromolecule, copolymerization of different components and the like have been

generally attempted in polyolefin resins or the like, but there has been no example in polyester resins. Furthermore, such a method is not preferable in that it changes inherent properties of the resins and is economically disadvantageous. Moreover, methods for improving printability, particularly printability with respect to ink for cellophane, include a method in which corona discharge treatment is performed on a film, a method in which a compound such as to render printing ink to readily adhere is coated on a film surface, and the like, but the former does not attain much effect of improving printability, and the latter requires an additional processing step and thus is economically disadvantageous.

General methods for giving antistatic properties to a film include a method in which an antistatic agent is coated on the film surface or blended in the film. However, in a case of polyester films, such a method has merits and demerits, and there has been almost no antistatic agent which gives satisfactory antistatic ability particularly under low-humidity conditions.

Thus, it has been very difficult to improve even one of various defects of polyester films as a packaging material, that is, lack of toughness such as flexing resistance and pinhole resistance, printability and antistatic properties with low dependence on humidity and it has been very difficult to improve these defects at the same time.

The present inventors have intensely studied on a method for overcoming the above defects of polyester films as a packaging material at the same time, and they have reached the present invention.

Specifically, the present invention relates to a polyester film having excellent processability obtained by melt-extruding: a polyester consisting of a residue of dibasic acids wherein at least 80% by mol of the dibasic acids is terephthalic acid and a residue of glycols; a block copolyester

consisting of a polyester segment having a high melting point and a soft polymer segment having a low melting point, said soft polymer segment having a low melting point being contained in an amount of 0.5 to 10% by weight on the basis of the whole weight of the polymer mixture; and a sulfonic acid metal salt derivative having a heat resistance such that the initial temperature showing a substantial decrease of the weight is 200°C or higher when it is heated in air, said sulfonic acid metal salt derivative being contained in an amount of 0.05 to 5% by weight with respect to the whole weight of the polymer mixture, and subsequently drawing at least uniaxially and then heat-treating the resultant at a temperature of 170°C or higher and lower than the melting point of the polyester.

The polyester of the present invention consists of a residue of dibasic acids wherein at least 80% by mol is terephthalic acid and a residue of glycols, which may be abbreviated to "terephthalic acid polyester" hereinafter. The dibasic acid residue comprises predominantly terephthalic acid residue, but 20% by mol or less thereof may be a residue of other dibasic acid. Residues of other dibasic acids include residues of isophthalic acid, phthalic acid, adipic acid, sebacic acid, succinic acid, oxalic acid and the like, and residues of oxy acids such as p-hydroxybenzoic acid may also be used.

The glycol residue may be a residue of normal alkylene glycols, and residues of ethylene glycol, propylene glycol, trimethylene glycol, tetramethylene glycol, cyclohexanedimethanol and the like can be exemplified. Practically, a residue of ethylene glycol or tetramethylene glycol is used.

The block copolyester used in the present invention is a copolymer consisting of a polyester segment having a high melting point and a soft polymer segment having a low melting point and a molecular weight of 400 to 8,000, and is a polymer consisting of constitutional units such that a polymer produced

by the components composing the polyester segment having a high melting point alone has a melting point of at least 170°C, and the soft polymer segment has a melting or softening point of 100°C or lower when measured for the components of the soft polymer segment alone. Components composing the polyester segment having high crystallinity and melting point are those which exhibit fiber-forming properties and has a melting point of 170°C when a polymer is produced by such components alone, and may be, for example, a polyester consisting of a residue of aromatic dicarboxylic acids, such as terephthalic acid, isophthalic acid, 1,5-naphthalenedicarboxylic acid, 2,6-naphthalenedicarboxylic acid and the like, and a residue of aliphatic, aromatic or alicyclic diols, such as ethylene glycol, propylene glycol, tetramethylene glycol, pentamethylene glycol, 2,2-dimethyltrimethylene glycol, hexamethylene glycol, p-xylylene glycol, cyclohexanedimethanol and the like; a copolyester containing a residue of oxy acids such as p-( $\beta$ -hydroxyethoxy)benzoic acid, p-hydroxybenzoic acid and the like in addition to the above-mentioned polyester, as a component to be copolymerized therewith; a polyamidoester consisting of a residue of aromatic ether dicarboxylic acids such as 1,2-bis(4,4'-dicarboxymethylphenoxy)ethane, or di(4-carboxyphenoxy)ethane and the like and a residue of the diols as mentioned above, and the like can be exemplified. Components composing the soft polymer segment having a low melting point and a molecular weight of 400 or more are those which are substantially in the non-crystalline state in the polyester block copolymer and have a melting or softening point of 100°C or lower when it is measured on the components composing the segment alone. The molecular weight of the soft polymer segment is usually 400 to 8,000. When the molecular weight is 400 or less, the block copolyester thus obtained has too low a melting point and shows too large an adhesion, which results



in difficulty in terms of processability in compounding into a film and further does not sufficiently give the desired toughness, such as flexing resistance and pinhole resistance, to the polyester. On the other hand, when the molecular weight is 8,000 or more, the phase of the non-crystalline polymer segment having a low melting point of the resulting block copolyester is separated, and as a result, the block copolyester shows an extremely high melting viscosity and becomes hard and brittle. Accordingly, after the copolymerization reaction, the block copolyester thus obtained can hardly be removed from the reaction vessel, and further when the block copolyester is blended in a film, the polyester film shows an inferior transparency. Thus, such a molecular weight is not preferable. Particularly preferred molecular weight of the soft polymer segment ranges from 700 to 6,000. A ratio of components composing the soft polymer segment having a low melting point in the polyester block copolymer ranges from 5 to 95% by weight, particularly preferably 10 to 90% by weight. The components composing the soft polymer segment having a low melting point may be a polyether, such as polyethylene oxide glycol, polypropylene oxide glycol, polytetramethylene oxide glycol, a glycol copolymer of ethylene oxide and propylene oxide, or a glycol copolymer of ethylene oxide and tetrahydrofuran, an aliphatic polyester, such as polyneopentyl azelate, polyneopentyl adipate, or polyneopentyl sebacate, or a polylactone, such as poly-ε-caprolactone or polypivalrolactone.

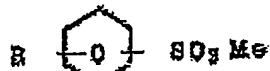
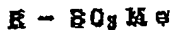
Specific examples of the block copolyester used in the present invention are polyethylene terephthalate-polyethylene oxide block copolymer, polytetramethylene terephthalate-polyethylene oxide block copolymer, polyethylene terephthalate-polytetramethylene oxide block copolymer, polytetramethylene terephthalate-polytetramethylene oxide block copolymer, polyethylene terephthalate-polyethylene oxide polypropylene

oxide block copolymer, polyethylene terephthalate-poly-ε-caprolactone block copolymer, polyethylene terephthalate-polypivrolactone block copolymer, polyethylene terephthalate-polyethylene adipate block copolymer, polyethylene terephthalate-polyneopentyl sebacate block copolymer, polytetramethylene terephthalate-polyethylene dodecanate block copolymer, polytetramethylene terephthalate-polyneopentyl dodecanate block copolymer, a block copolymer of a polyester made from di(4-carboxyphenoxy)ethane and ethylene glycol with polyethylene glycol, or a block copolymer of a polyester made from bis(N-p-carboxyethoxyphenyl)adipamide and ethylene glycol with polyethylene glycol.

In the mixture of this polyester block copolymer in the polyester film, the soft polymer segment having a low melting point is totally contained in the range of 0.5 to 10% by weight, preferably 0.5 to 5% by weight on the basis of the whole weight of the mixed polymer forming the film. When the content is less than 0.5% by weight, it does not sufficiently show the desired effect, and on the other hand, when the content is more than 10% by weight, the effect thereof shows little increase in result and shows an adverse result, such as the decrease of the transparency and other physical properties of the film.

The other component of the present invention: sulfonic acid metal salt derivative compound having a heat resistance such that the initial temperature showing a substantial decrease of the weight is 200°C or higher when it is heated in air is a sulfonic acid metal salt derivative in which a weight decrease of the compound substantially initiates at 200°C or higher after passing the induction period for the weight decrease, when the weight decrease property on heating is measured with a thermobalance, while increasing temperature in the air, as shown in Figure 1. An example of such a sulfonic acid metal salt derivative may be a compound having a structure

as shown below:



wherein R is an alkyl group having 8 to 20 carbon atoms, and may be octyl, nonyl, decyl, undecyl, dodecyl, octadecyl, or a mixture thereof; and Me is an alkali metal or an alkaline earth metal, and may be sodium or potassium as an alkali metal, and calcium, barium or magnesium as an alkaline earth metal. Specific examples of such a compound include sodium octylsulfonate, sodium decylsulfonate, sodium dodecylsulfonate, potassium dodecylsulfonate, sodium salt of a mixture of alkylsulfonic acids having an average carbon number of 14, sodium octylbenzenesulfonate, sodium nonylbenzenesulfonate, sodium dodecylbenzenesulfonate, potassium dodecylbenzenesulfonate, sodium nonylnaphthalenesulfonate, sodium dodecylnaphthalenesulfonate, potassium dodecylnaphthalenesulfonate and the like.

Such a sulfonic acid metal salt derivative compound having a heat resistance such that the initial temperature showing a substantial decrease of the weight is 200°C or higher when it is heated in air may be used alone or in a combination with two or more thereof, and a mixing ratio in a film is 0.05 to 5% by weight, preferably 0.1 to 3% by weight, on the basis of the whole weight of the mixed polymer. The effect of the present invention, particularly the effects of exhibiting antistatic properties with low dependence on humidity, can be sufficiently obtained by addition of the compound in a comparatively small amount. When the compound is added in an amount larger than 5% by weight, the effect thereof shows little

increase in result and shows an adverse result, such as the decrease of the transparency and the decrease of the physical properties owing to the promotion of the heat deterioration of the polyester film. Thus, such an amount is not preferable.

In production of the film of the present invention, examples of methods for blending of the block copolyester and the sulfonic acid metal salt derivative compound having a particular heat resistance to the terephthalic polyester is, but not particularly limited to, adding the block copolyester and the sulfonic acid metal salt compound to the polymerization system of the terephthalic polyester when the polymerization is finished, or supplying constant amounts of the terephthalic polyester, the block copolyester and the sulfonic acid metal salt compound to an inlet for supplying a resin of an extruder and then melt-kneading the mixture. In the present invention, other additives, for example, a surfactant, ultraviolet absorber, various stabilizers, lubricant, fire retardant, pigment, dyestuff, or the like may also be added, if necessary, in addition to the block copolyester and the sulfonic acid metal salt compound.

The mixed polymer thus blended can then be formed into a film by the normal polyester film-forming methods, for instance, T-die method, or inflation method, by which a non-drawn film is formed. The effects of the present invention can be exhibited more effectively by a drawn film obtained by drawing the non-drawn film at least uniaxially, preferably biaxially.

The drawing may be carried out under similar conditions as those in the drawing of the terephthalic polyester, but when the block copolyester and the sulfonic acid metal salt compound are blended in a high ratio, the conditions may be varied appropriately. The drawing temperature is preferably 60°C to 100°C. This drawing is an important factor for obtaining the desired effects of the present invention, and the film should

be at least uniaxially drawn and it is particularly desirable to draw biaxially in a rectangular direction to each other. The draw ratio is not specifically limited, but it is usually 1.2 to 6 times, preferably 1.5 to 6 times, in case of the uniaxial drawing, and is perpendicularly 1.2 to 6 times and laterally 1.2 to 6 times in case of the biaxial drawing.

After the film formation and the drawing thereof, it is essential for the present invention to subject the resultant to the heat treatment. The heat treatment may be carried out at a temperature of 170°C or higher and below the melting point of the terephthalic polyester for 0.1 second to 5 minutes. It is more efficient to carry out the heat treatment at a higher temperature region since the treating time can be reduced. The heat treatment may be carried out by exposing the film into an atmosphere set at the above temperature conditions or by contacting the film with a roller heated at the above temperature, but is not limited thereto. Besides, the heat treatment may be carried out at the shrunk state, normal state or pulled state of the film, but particularly, in case of treatment at the shrunk state, the relaxation ratio is preferably within 50%, and in case of treatment at the pulled state, the pulling ratio is preferably within 150%.

The polyester film obtained in accordance with the present invention has excellent flexing resistance, pinhole resistance, and printability with a printing ink for cellophane films and further exhibits extremely excellent antistatic properties with low dependence on humidity, and has these properties which are defects of conventional polyester films as a packaging material sufficiently improved.

The present invention is specifically illustrated by the following Examples, but of course, the present invention is not limited only thereto.

The values of characteristics in the present invention are measured as follows.

1) Pinhole resistance:

A film is fixed onto the top of a glass tube having a diameter of 10 mm in a loose bag-like shape and the glass tube is given by an air pressure of 1 kg/cm<sup>2</sup> and then a vacuum, which operations are alternately repeated in the rate of 10 times/minute. Pinhole resistance is shown by the number of the operations till pinholes occur in the film.

2) Printability:

A film is printed with an ink for cellophane (white) by using a gravure printing machine. Thereafter, a tape is adhered on the printing layer, and then the tape is rapidly peeled off. Printability is shown by the rate (%) of the area of remaining printing ink on the film.

3) Antistatic properties:

It is shown by a half-life time of charged voltage measured at 20°C and a relative humidity (RH) of 65% and 40% using Static Honest Meter (made by Shishido Shokai). In charge of voltage, 10,000 V was charged on the test sample from the height of 15 mm.

4) The initial temperature showing a decrease of the weight of antistatic agent:

The antistatic agent is heated in air at a raising rate of 4°C/minute and the weight decrease of the agent is measured by a TM-2 type thermobalance made by Shimazu Seisakusho. The initial temperature is shown at the point of 10% decrease (X point) in the relation between the weight decrease ratio and the temperature.

EXAMPLE 1

Using a mixture solution of phenol and tetrachloroethane (phenol/tetrachloroethane = 6/4), polyethylene terephthalate having intrinsic viscosity of 0.62 as measured at 30°C; block copolyester obtained by polymerizing 400 parts of bishydroxyethyl terephthalate, 400 parts of polyethylene oxide

glycol with a molecular weight of 4000, 0.4 part of antimony trioxide and 0.4 part of zinc acetate by a polymerization method similar to a polymerization method used for the production of normal terephthalic polyester (2.0, 3.0 or 4.0% by weight as an amount of polyethylene oxide glycol segment in the whole mixed polymer); and sodium dodecylbenzenesulfonate (1.0% by weight) were mixed together by a blender. Thereafter, the mixture was melt-extruded by an extruder equipped with T-die (diameter of screw: 20 mm), and then cooled with a cooling roll at 80°C to give a non-drawn film with a thickness of 250 $\mu$ . The non-drawn film was then drawn 3.5 times perpendicularly at 90°C and then 3.5 times laterally at 90°C, and the resultant was set by heating at 200°C for 30 seconds. For the comparison purpose, the polyethylene terephthalate alone used above was melt-extruded, drawn biaxially and further subjected to the heat set under the same conditions. The flex pinhole resistance, printability and antistatic properties under conditions at various humidity of these films are shown in the following Table 1.

It was found that the film in accordance with the present invention obtained by mixing a block copolyester and sulfonic acid metal salt compound having a particular heat resistance and drawing and heat-treating the mixture had excellent flex pinhole resistance, printability with an ink for cellophane, and antistatic properties at a low humidity.

TABLE 1

	Amount added of block copolyester (% by weight)*	Amount added of sodium dodecylbenzene sulfonate (% by weight)	pinhole resistance (times)	printability (%)	Half-life period of the charged voltage (second)	
					65% RH	40% RH
Comparative Examples	0	0	300	0	>400	>400
Examples of the present invention	2.0	1.0	600	100	2.0	3.0
	3.0	1.0	480	100	1.5	2.0
	4.0	1.0	450	100	1.5	1.5

\*It is shown by the content of the polyethylene oxide glycol in the whole mixed polymer from which the film is formed.



## EXAMPLE 2

Characteristics of films obtained by adding block copolyester and sulfonic acid metal salt compound as shown in Tables 2 and 3 in the same manner as in Example 1 and biaxially drawing and heat-treating the mixture are shown in Table 4.

For comparison purpose, some films were produced in the same method while blending a block copolyester alone and sulfonic acid metal salt compound alone, and properties thereof were evaluated.

As is made clear from Table 4, it was found that only the film of the present invention improved the desired flex pinhole resistance, printability, antistatic properties in low humidity.

TABLE 2: Block copolyester

No.	Polyester segment having a high melting point		Non-crystalline segment having a low melting point		
	Structure	Ratio of copolymerization (% by weight)	Structure	Molecular weight	Ratio of copolymerization (% by weight)
I	Polyethylene terephthalate	30	Polyethylene oxide glycol	2000	70
II	Polyethylene terephthalate	30	Polyethylene oxide glycol	4000	70
III	Polyethylene terephthalate	50	Polyethylene oxide glycol	2000	50
IV	Polytetramethylene terephthalate	60	Polytetramethylene oxide glycol	1500	40
V	Polytetramethylene terephthalate	70	Polytetramethylene oxide glycol	1500	30

TABLE 3: Sulfonic acid metal salt compound

No.	Structure	Initial temperature of weight decrease (°C)*
VI	$C_{12}H_{25}OSO_3Na$	150
VII	$C_{17}H_{35}O(CH_2CH_2O)_2SO_3Na$	170
VIII	$C_{17}H_{35}SO_3Na$	260
IX	R $SO_3Na$ (R is a mixture of alkyl groups of an average carbon number of 15)	250
X	R $SO_3Na$ (R is a mixture of alkyl groups of an average carbon number of 15)	210

\*It is the initial temperature of weight decrease measured with a thermobalance as shown in Figure 1.

TABLE 4

	Block copolyester		Sulfonic acid metal salt compound		Pinhole resistance (times)	Printability (%)	Half-time period of charged voltage (second)	
	Compound	Amount* (% by weight)	Compound	Amount (% by weight)			65% RH	40% RH
Comparative Examples	I	3.0	-	-	450	100	180	>400
	II	3.0	-	-	450	100	150	>400
	III	3.0	-	-	450	100	180	>400
	IV	3.0	-	-	500	100	>400	>400
	V	3.0	-	-	500	100	>400	>400
	-	-	VI	1.0	200	0	>400	>400
	-	-	VII	1.0	200	0	>400	>400
	-	-	VIII	1.0	200	0	3.0	>400
	-	-	IX	1.0	200	0	5.0	>400
	-	-	X	1.0	200	0	10.0	>400
	I	3.0	VI	1.0	200	100	180	>400
	I	3.0	VII	1.0	200	100	180	>400
Present invention	I	3.0	VIII	1.0	450	100	1.5	2.0
	I	3.0	IX	1.0	450	100	1.5	2.0
	I	3.0	X	1.0	450	100	1.5	2.0
	II	3.0	VIII	1.0	450	100	1.5	2.0
	III	3.0	IX	1.0	450	100	1.5	2.0
	VI	3.0	VIII	1.0	500	100	1.5	10.0
	V	3.0	IX	1.0	500	100	1.5	10.0

\*It is shown by the content of the noncrystalline polymer segment component having a low melting point in the whole mixed polymer from which the film is formed.

### EXAMPLE 3

To polyethylene terephthalate used in Example 1, the compound No. I in Table 2 and the compound No. VIII in Table 3 were added in an amount of 3% by weight and 1% by weight, respectively as a content of polyethylene oxide glycol in the whole film mixture. The mixture was biaxially drawn in the same manner as described in Example 1. The drawn film was treated under various heat treatment conditions and then the characteristics of the resulting films were measured. The results are shown in Table 5.

TABLE 5

	Temperature for heat treatment (°C) x 30 seconds	Flex pinhole resistance (times)	Printability (%)	Half-life period of the charged voltage (second)	
				65% RH	40% RH
Comparative Examples	100	450	100	>400	>400
	140	450	100	>400	>400
Present Invention	180	450	100	3.0	3.0
	210	450	100	1.5	2.0
	230	450	100	1.5	2.0

As is made clear from Table 5, the films heat-treated by the method of the present invention had excellent flex pinhole resistance, printability and antistatic properties with low dependence on humidity.

#### 4. Brief Description of the Drawing

Figure 1 is a graph showing characteristics of weight decrease on heating in air of antistatic agents VI to IX used in Example 2 (Table 3) of the present invention as measured with

a thermobalance and an initial temperature showing a substantial decrease of the weight-a temperature showing 10% weight decrease of each compound.

Figure 1

